# PATENT ABSTRACTS OF JAPAN

(11)Publication number:

01-263910

(43)Date of publication of application: 20.10.1989

51)Int.CI.

G11B 5/66

G11B 5/704

G11B 5/706

21)Application number: 63-089058

(71)Applicant: HITACHI LTD

22)Date of filing:

13.04.1988

(72)Inventor: MATSUDA YOSHIFUMI

SHIROISHI YOSHIHIRO

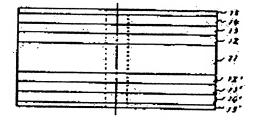
HISHIYAMA SADAO SUZUKI HIROYUKI OONO TADAYUKI SAITO SHINICHIRO TAKAGI KAZUMASA

# 54) PERPENDICULAR MAGNETIC RECORDING MEDIUM AND MAGNETIC DISK DEVICE

## 57)Abstract:

URPOSE: To improve magnetic characteristics and adhesiveness so asobtain a high recording density and to improve reliability by using itermediate layers of a Ti-alloy contg. specific elements in addition to Ti s nonmagnetic intermediate layers.

ONSTITUTION: The thin films added with at least one kind of the lement selected from the group consisting of V, Nb, Ta, Cr, Mo, W, Mn, Ni, d, Pt, Cu, Ag, Au, C, Si, Ge, Ru, Os, Rh, and Ir are used for the termediate layers 13, 13' of the Ti-base alloy of the perpendicular agnetic recording medium provided with the intermediate layers 13, 13' f the Ti-base alloy between hexagonal magnetic alloy layers 14, 14' and a onmagnetic substrate 11. The c-axis orientational property and magnetic naracteristics of the hexagonal magnetic alloy layers 14, 14' are thereby reatly improved and the magnetic disk and device having the good scording and reproducing characteristics and reliability are thereby obtd.



#### **:GAL STATUS**

)ate of request for examination]

)ate of sending the examiner's decision of rejection] find of final disposal of application other than the caminer's decision of rejection or application converted gistration]

Date of final disposal for application]

Patent number]

Date of registration]

Number of appeal against examiner's decision of

ejection]

Date of requesting appeal against examiner's decision

f rejection]

Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

#### \* NOTICES \*

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

# **CLAIMS**

# (57) [Claim(s)]

[Claim 1] The vertical-magnetic-recording medium characterized by having Ti radical alloy interlayer containing at least one sort of elements chosen from the group which consists of V, Nb, Ta, Cr, Mo, W, Mn, nickel, Pd, Pt, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, and Ir besides Ti at least between the magnetic alloy layer of hexagonal system, and a nonmagnetic substrate. [Claim 2] A vertical-magnetic-recording medium given in the 1st term of a patent claim characterized by preparing a high permeability magnetic layer further between Ti radical alloy interlayers and the nonmagnetic substrates containing at least one sort of elements chosen from the group which consists of V, Nb, Ta, Cr, Mo, W, Mn, nickel, Pd, Pt, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, and Ir besides Above Ti.

[Claim 3] A vertical-magnetic-recording medium given in the 1st term of a patent claim or the 2nd term characterized by the presentation ratio being less than [ more than 1at%25at% ] in a total amount including at least one sort of elements chosen from the group which consists of V, Nb, Ta, Cr, Mo, W, Mn, nickel, Cu, C, Si, and germanium to said Ti radical alloy interlayer. [Claim 4] A vertical-magnetic-recording medium given in the 3rd term of a patent claim characterized by the presentation ratio of any or one sort of elements being less than [ more than 5at%20at% ] among Nb and Ta to said Ti radical alloy interlayer.

[Claim 5] A vertical-magnetic-recording medium given in the 1st term of a patent claim or the 2nd term characterized by the presentation ratio being less than [ more than 0.1at%10at% ] in a total amount including at least one sort of elements chosen from the group which consists of Ru, Os, Rh, Ir, Pd, Pt, Ag, and Au to said Ti radical alloy interlayer.

[Claim 6] A vertical-magnetic-recording medium given in the 5th term of a patent claim characterized by the presentation ratio being less than [ more than 1at%7.5at% ] in a total amount including at least one sort of elements chosen from the group which consists of Ru, Os, Rh, Ir, Pd, Pt, Ag, and Au especially to said Ti radical alloy interlayer.

[Claim 7] A vertical-magnetic-recording medium given in the 1st term of a patent claim thru/or the 6th term characterized by Co presentation ratio using the magnetic alloy layer of said hexagonal system as Co radical alloy thin film beyond 50at%.

[Claim 8] A vertical-magnetic-recording medium given in the 7th term of a patent claim characterized by the presentation ratio being less than [ more than 0.1at%25at% ] in a total amount including at least one sort of elements with which said Co radical alloy thin film was chosen from the group which consists of Cr, V, Mo, W, Ti, Mn, Re, Sm, Fe, and O. [Claim 9] at least one sort of elements with which said Co radical alloy thin film was chosen from the group which consists of Zr, Ti, Hf, Ta, Ru, Rh, Pd, and Pt — containing — the presentation ratio — a total amount — less than [ more than 0.1at%15at% ] — the claim characterized by containing — a vertical-magnetic-recording medium given in the 7th term or the 8th term. [Claim 10] A vertical-magnetic-recording medium given in the 1st term of a patent claim thru/or the 9th term characterized by using any one sort of a tempered glass substrate, aluminum alloy substrate with a nickel-P deposit, and the ceramic substrate as said nonmagnetic substrate. [Claim 11] The magnetic disk drive using a vertical-magnetic-recording medium given in the 1st term of a patent claim thru/or the 10th term.

[Translation done.]

### \* NOTICES \*

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.

2.\*\*\*\* shows the word which can not be translated.

3.In the drawings, any words are not translated.

### **DETAILED DESCRIPTION**

[Detailed Description of the Invention]

[Industrial Application]

This invention relates to the suitable medium for magnetic disk drives especially for high density record with respect to the vertical-magnetic-recording medium which attracts attention as a medium in which next-generation super-high density record is possible.

[Description of the Prior Art]

Conventionally, the medium using the Co-Cr alloy thin film as a magnetic film is proposed like the publication to JP,57-17282,B as a vertical-magnetic-recording medium for super-\*\*\*\*\* record. There are vacuum deposition, the sputtering method, plating, etc. as a method of forming the medium for vertical magnetic recordings. Recently, improvement in recording density is required increasingly, therefore Ti interlayer is prepared between a Co-Cr alloy thin film and a substrate like JP,49-74912,A and JP,58-14318,A, and the proposal which controls crystallinity and acquires a high c-axis stacking tendency is made. moreover, to JP,58-133624,A In order to lessen the difference of coefficient of thermal expansion with a Co-Cr alloy and to make small the truck gap at the time of record and playback, as a substrate for vertical-magnetic-recording media By using Ti radical alloy or preparing Ti alloy interlayer in JP,62-143227,A, 62-143228, and 62 No. -143229 official report as a substrate layer further, and setting the diameter of crystal grain of a magnetic layer to 10-300nm further The proposal which is going to obtain the medium of high coercive force also with a vacuum deposition method is also made.

[Problem(s) to be Solved by the Invention]

However, most of these invention is seldom examined in the rigid disk for computers using the substrate which consists of a flat and hard ingredient like tempered glass or a nickel-P plating aluminum alloy about the tape for magnetic recording and flexible disk by vacuum deposition, the ion plating method, etc. Then, this invention persons formed the direct Co-Cr alloy by the RF sputtering method on the nickel-P plating aluminum alloy substrate first, and evaluated the property. The presentation of Cr was made into substrate temperature:room temperature -150 degree C at the time of membrane formation, the Ar gas pressure 3 - 30mTorr (0.4-4Pa), the injection power flux density 1 - 10 W/cm2, and 60-250nm of thickness 5 - 25at% here. Any Co-Cr film of vertical coercive force was as low as 300 or less Oes, moreover, the distribution deltatheta50 of the c-axis stacking tendency of Co-Cr was also as bad as 10 degrees or more, and good perpendicular magnetic anisotropy films were not obtained. The same was said of the tempered glass substrate. This is because the high orientation of the c-th page which is the maximum \*\*\*\* of Co-Cr with it is checked compared with an organic system substrate usual in a nickel-P substrate or a tempered glass substrate. [ high surface energy and ] [ hexagonal ] In order to attain high recording density in the magnetic film for vertical magnetic recordings generally, while heightening vertical coercive force, it is known that especially the thing done at right angles to a film surface for the orientation of the crystallographic axis (c-axis) of perpendicular magnetic anisotropy films is important.

The purpose of this invention forms the perpendicular magnetic anisotropy films which have a crystal stacking tendency good also as a rigid disk using hard substrates, such as a tape for magnetic recording, and not only a flexible disk but a tempered glass metallurgy group ingredient,

magnetic properties, and adhesion by the sputtering method high adhesion is acquired etc., and high recording density is possible for it, and it is to offer a reliable magnetic-recording medium. [The means for solving a technical problem]

Among nonmagnetic substrates, such as a magnetic alloy layer of hexagonal system, a tempered glass substrate, aluminum alloy substrate with a nickel-P deposit, and a ceramic substrate, Si radical alloy, The medium which prepared various nonmagnetic metatarsus layers, such as Ti radical alloy and Zr radical alloy, is formed by a spatter etc. According to research of this invention person who examined dependability, such as adhesion and corrosion resistance, etc. wholeheartedly to the crystallographic property [ of the medium ] and magnetic property, and record reproducing-characteristics pan The above-mentioned purpose by using Ti radical alloy interlayer containing at least one sort of elements chosen from the group which consists of V. Nb, Ta, Cr, Mo, W, Nm, nickel, Pd, Pt, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, and Ir besides Ti as said nonmagnetic interlayer It is attained. As for that presentation ratio, less than [ more than 1at%25at%] is desirable in a total amount including at least one sort of elements chosen from the group which consists of V, Nb, Ta, Cr, Mo, W, Mn, nickel, Cu, C, Si, and germanium to this Ti radical alloy interlayer. It is desirable for the presentation ratio to consider as less than [ more than 0.1at%10at%] in a total amount including at least one sort of elements chosen from the group which consists of Pd, Pt, Ag, Au, Ru, Os, Rh, and Ir. Furthermore, it is more desirable for the presentation ratio of any or one sort of elements to be less than [ more than 5at%20at% ] especially among Nb and Ta to said Ti radical alloy interlayer, and it is more desirable for the presentation ratio to consider as less than [more than 1at%7.5at%] in a total amount including the element chosen from the group which consists of Pd, Pt, Ag, Au, Ru, Os, Rh, and Ir. moreover -- as the magnetic alloy layer of said hexagonal system -- Co -- more than 50at% -at least one sort of elements chosen from the group X which contains and consists of Cr. V. Mo. W, Ti, Mn, Re, Sm, Fe, and O further -- a total amount -- less than [ more than 0.1at%25at% ] -if the included Co-X binary-system-alloy thin film is used, it is more effective on magnetic properties. furthermore, at least one sort of elements chosen from the group Y which consists of Zr, Ti, Hf, Ta, Ru, Rh, Pd, and Pt as a magnetic alloy layer of said hexagonal system -- a total amount -- less than [ more than 0.1at%15at% ] -- the included Co-Y binary-system-alloy thin film may be used. at least one sort of elements further chosen as said Co-X binary system alloy from said Y -- a total amount -- less than [ more than 0.1at%25at% ] -- if the included Co-X-Y alloy thin film of 3 yuan is used, it is more effective on corrosion resistance. [Function]

The above-mentioned means is based on the following operations. Raising the crystal stacking tendency of a Co-Cr magnetic film to JP,58-159225,A, 59-22236, 59-22225, 59 No. -33628 official report, etc. until now by forming a Co-Cr thin film through Ti thin film on heat-resistant substrates, such as polyimide, is known. Then, it examined first whether the same effectiveness would be acquired also to a nickel-P substrate. That is, nickel-P was plated, Ti interlayer of 20-600nm of thickness was formed by the RF sputtering method as substrate temperature:room temperature -200 degree C, the Ar gas pressure 3 - 30mTorr (0.4-4Pa), and the injection power flux density 1 - 10 W/cm2 on aluminum alloy substrate which carried out mirror polishing of the front face, the Co-Cr alloy magnetic layer of 60-250nm of thickness was formed further continuously, and the property was evaluated. Cr presentation was changed and considered to 5 - 25at%. Consequently, although the coercive force of Co-Cr became high by preparing Ti interlayer at 300 or more Oes and such high coercive force that substrate temperature is high was acquired, deltatheta50 which shows distribution of the c-axis stacking tendency of Co-Cr also in which film was as large as 10 degrees or more, and a good c-axis stacking tendency was not shown. In vacuum deposition, only the film of 15 degrees or more and a still lower stacking tendency was obtained, but only the same result as a nickel-P substrate was obtained also with the tempered glass substrate. On the other hand, organic system substrates, such as polyethylene terephthalate, polyimide, and POIAMIDO, were received, and the film with which deltatheta50 has a high c-axis stacking tendency with about 5 degrees was obtained also by the approach of a gap. A nickel-P substrate and a tempered glass substrate have high surface energy compared with an organic system substrate, and this is because the orientation of the c-

th page which is Ti interlayer's maximum \*\*\*\* will also be checked. Then, this invention persons are the above-mentioned membrane formation conditions about raising an interlayer's stacking tendency first, and decided to examine wholeheartedly the interlayer who added the element of 4A, 5A, 6A, 7A, 8, 1B, 2B, 3B, 4B and 5B, and 6B group to Ti by membrane formation and carrying out characterization. Consequently, Ti radical alloy interlayer containing at least one sort of elements chosen from the group which consists of V, Nb, Ta, Cr, Mo, W, Mn, nickel, Pd, Pt, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, and Ir besides Ti in which membrane formation conditions Compared with Ti single phase film, it became clear with RHEED, an X-ray diffraction method, etc. that it is easy to carry out orientation of the c-axis perpendicularly on a nonmagnetic substrate. Although the c-axis stacking tendency of the Co-Cr magnetism alloy layer formed on it with Ti radical alloy interlayer who added to Ti at Nb and Ta formed on the nickel-P substrate is actually shown in the 4th drawing 4 Fig. (a) To Ti radical alloy interlayer, when any one sort of presentation ratios of Nb and Ta are less than [more than 5at%20at%], deltatheta50 of 002 reflection of Ti-Nb, a Ti-Ta alloy interlayer, and a Co-Cr magnetism alloy layer is small, and it turns out especially that the c-axis stacking tendency of a Co-Cr magnetism alloy layer is high. The same effectiveness was accepted with the addition with the same said of the alloy which consists of the cases where one sort of the element chosen from the group which consists of a Nb-Ta alloy, or V, Cr, Mo, W, Mn, nickel, Cu, C, Si and germanium is added to Ti, or such combination. The film which added any one sort of Nb and Ta to Ti here has highly desirable corrosion resistance especially as an interlayer compared with the case where one sort of V, Cr, Mo, W, nickel, Cu, C, Si, and germanium is added. moreover, Ti radical alloy interlayer -- any one sort of Nb and Ta -- less than [ more than 5at%20at% ] -- when it added, the highest c-axis stacking tendency was acquired. Furthermore, to Ti radical alloy, although the c-axis stacking tendency of the Co-Cr magnetism alloy layer formed on Ti radical alloy interlayer who added Pt and Pd to Ti is shown in Fig. 4 (b), when any one sort of presentation ratios of Pt and Pd are less than [ more than 1at%7.5at% ], it turns out especially that the c-axis stacking tendency of a Co-Cr magnetism alloy layer is high. Also when the alloy of Ru, Os, Rh, Ir, Ag, Au, Pt, and Pd was added to the case where at least one sort of the element chosen from the group which consists of Ru, Os, Rh, Ir, Ag, and Au is added to Ti, or Ti, the same effectiveness was accepted with the same addition. Here, compared with Ti radical alloy system which added V, Nb, Ta, Cr, Mo, W, Mn, nickel, Cu, C, Si, and germanium to Ti, although these alloy systems are expensive, its corrosion resistance is high and is more desirable in respect of dependability. Anyway, by adding to Ti at least one sort of elements chosen from the group which consists of V, Nb, Ta, Cr, Mo, W, Mn, nickel, Pd, Pt, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, and Ir in this way That the c-axis stacking tendency as a Ti radical alloy interlayer improves Solid-solution limits, such as V, Nb, Ta, Cr, Mo, W, Mn, nickel, Pd, Pt, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, Ir, etc. which can hold the hexagonal phase of Ti in the state diagram of the bulk of Ti radical alloy, are very small. Therefore, it is easy to segregate components, such as V, Nb, Ta, Cr, Mo, W, Mn, nickel, Pd, Pt, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, and Ir, to the grain boundary of Ti radical alloy interlayer's columnar crystal. Therefore, it is for the c-th page which is the maximum \*\*\*\* to tend to grow in parallel with a substrate side. however, it is difficult for differing from the state diagram of bulk remarkably to usually come out of the absolute value of a presentation, a phase condition, etc., and for the state diagram in the thin film condition which generally formed membranes by the sputtering method etc. to expect this effectiveness conversely only from the state diagram of bulk, and it needs a detailed experiment which was already explained. Furthermore, Co-Cr, Co-V, Co-Mo, Co-W, Co-Re, Co-Ti, Co-Sm, Co-Mn, Co-Ta, Co-Zr, Co-Hf, Co-Pd, Co-Pt, Co-Fe, Co-O, Co-Cr-Rh, Co-Cr-Ru, Co-Cr-Ta, Since hexagonal system magnetism alloys, such as Co-Cr-Zr, Co-Cr-Pt, Co-Cr-Pd, Co-Cr-Ti, Co-Cr-Hf, and Co-Ti-Ta, have the above-mentioned Ti radical alloy and the near lattice constant, The perpendicular magnetic anisotropy films which show the high c-axis stacking tendency to which a hexagonal system magnetism alloy layer tends to grow on Ti radical alloy, and changes from the above-mentioned hexagonal system magnetism alloy in epitaxial will be obtained. Since corrosion resistance is high compared with Co radical binary system alloy, Co radical alloy of 3 yuan is desirable here.

Next, the magnetic properties of the high orientation perpendicular magnetic anisotropy films which consist of this invention, and record reproducing characteristics are described. The magnetic properties of the Co-Cr magnetism alloy layer formed on Ti radical alloy interlayer who added Nb, Ta, and Pt and Pd to Ti, respectively were shown in Fig. 4 (a) and Fig. 4 (b). The perpendicular magnetic properties which were [ be / a perpendicular magnetic anisotropy is high and / a remanence ratio / large ] excellent in Ti radical alloy presentation with the high c-axis stacking tendency of a Co-Cr magnetism alloy layer were acquired. Moreover, when at least one sort of elements chosen from the group which consists of V, Cr, Mo, W, Mn, nickel, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, and Ir were added to Ti, the perpendicular temperament property excellent in Ti radical alloy presentation with the high c-axis stacking tendency of Co radical magnetism alloy layer was acquired similarly. Better record reproducing characteristics are shown, at least one sort of alloying elements especially chosen from the group X which consists of Cr, V, Mo, W, Ti, Mn, Re, Sm, Fe, and O as a magnetic alloy layer of said hexagonal system -a total amount -- less than [ more than 0.1at%25at% ], if included Co radical alloy thin film is used When the amount of alloying elements was made into less than [ 0.1at% ], the record component within a field became remarkably strong, when it was made [ more ] than 25at%, the playback output declined remarkably, and as for record reproducing characteristics, all deteriorated, furthermore, at least one sort of alloying elements chosen from the group Y which consists of Zr, Ti, Hf, Ta, Ru, Rh, Pd, and Pt as a magnetic alloy layer of said hexagonal system -- a total amount -- less than [ more than 0.1at%15at% ], although good record reproducing characteristics are shown even if it uses included Co radical alloy thin film Even if it made the amount of alloying elements into less than [ 0.1at% ] and made [ more ] it than 15at%, record reproducing characteristics deteriorated like the above. at least one sort of elements chosen as the Co-X binary system alloy from Y group as mentioned above here -- a total amount -- less than [more than 0.1at%25at%] -- since a playback output shows the corrosion resistance which was excellent although it fell a little, added Co-X-Y the alloy of 3 yuan has it. [ more desirable than an application top ] Although the above effectiveness was checked also by vacuum deposition, about 7 degrees and the magnitude of effectiveness of deltatheta50 were large compared with what is depended on the sputtering method. The film by which this was generally formed with vacuum deposition is because the kinetic energy of a vacuum evaporationo particle is inferior to a membranous stacking tendency and adhesion compared with the film by the sputtering method which has kinetic energy high about single figure with 0.1-1eV since it is small. Thus, since the film by the sputtering method is excellent also in adhesion, it is desirable also in respect of dependability, such as sliding-proof nature. Even if the above effectiveness added the impurity gas of H2, O2, and N2 grade to 1vol% in the gas under sputtering, it was almost the same.

The above effectiveness, by the sputtering method etc., on a nonmagnetic substrate as a nonmagnetic interlayer By forming Ti radical alloy interlayer containing at least one sort of elements chosen from the group which consists of V, Nb, Ta, Cr, Mo, W, Mn, nickel, Pd, Pt, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, and Ir Since adhesion is also high while a hexagonal system magnetism alloy layer shows the perpendicular magnetic properties which were perpendicularly high, and were excellent in it to the substrate side, [ of the c-axis stacking tendency ] By using the hexagonal system magnetism alloy layer which consists of this invention, the vertical—magnetic-recording medium and equipment which have especially excellent record reproducing characteristics and dependability can be offered.

[Example]

Hereafter, the example of this invention is explained.

[Example 1]

The nonmagnetic deposit which consists of the alloy with which nonmagnetic substrate [ with which 11 consists of aluminum alloy etc. ], 12, and 12' makes a main component nickel-P, nickel-W-P, or these in Fig. 1, The hexagonal system magnetism alloy layer to which nonmagnetic interlayer [ to whom 13 and 13' changes from Ti radical alloy ], 14, and 14' changes from Co-Cr, 15 and 15' is a protection lubricating layer which consists of C, B, B4C, Si-C, Co3O4, SiO2, Si3N4, W-C, Si-C, Zr-C, etc., and each is formed as shown below. After forming the nonmagnetic

12wt%P-nickel deposit 12 of 20 micrometers of thickness, and 12' on outer-diameter 130mmphi, bore 40mmphi, and aluminum alloy substrate 11 with a thickness of 1.9mm, mirror polishing of the front face was carried out so that it might become 10nm of center line average side granularity of owner Perilla frutescens (L.) Britton var. crispa (Thunb.) Decne. about irregularity detailed to a circumferencial direction, and thickness was set to 15 micrometers. After forming Ti radical alloy interlayer of the following [ the spatter conditions of the substrate temperature of 100 degrees C, 0.7Pa of Ar gas pressure, and RF injection power 5 W/cm2 ] 400nm of thickness with RF magnetron sputtering equipment on this substrate and considering as the nonmagnetic interlayer 13 and 13', the 21at%Cr-Co magnetic layer 14 and 14' were formed 250nm of thickness on the same spatter conditions. As a nonmagnetic interlayer here to Ti radical alloy interlayer V, Nb, Ta, Cr, Mo, W, Mn, nickel, Cu, C, Si, and germanium -- each 10at% -- with the thin film added and formed Pd, Pt, Ag, Au, Ru, Os, Rh, and Ir -- each 5at% -- with DC magnetron sputtering equipment to the pan using the thin film added and formed The protection lubricating layer 15 and 15' which consist of C on the spatter conditions of the substrate temperature of 100 degrees C, 1.3Pa of Ar gas pressure, and DC injection power 3 W/cm2 were formed 30nm of thickness, and the magnetic disk was produced.

[Example 2]

In Fig. 2, nonmagnetic substrate [ which consists of the tempered glass which, as for 21, set center line average side granularity of owner Perilla frutescens (L.) Britton var. crispa (Thunb.) Decne. to 5nm for irregularity detailed to a circumferencial direction on the front face ] 22, nonmagnetic interlayer [ to whom 22' changes from Ti radical alloy ] 23, hexagonal system magnetism alloy layer [ to which 23' changes from Co-Cr etc. ], 24, and 24' is a protection lubricating layer which consists of C, B, B4C, Si-C, etc., and each is formed, as shown below. On the tempered glass substrate 21, after forming Ti radical alloy interlayer of the following [ the spatter conditions of 2 ] 200nm of thickness the substrate temperature of 100 degrees C. 0.5Pa of Ar gas pressure, and 7W [/cm ] DC injection power and considering as the nonmagnetic interlayer 22 and 22' with DC magnetron sputtering equipment, the 20at%Cr-Co magnetic layer 23 and 23' were formed 200nm of thickness on the same spatter conditions. here -- Ti radical alloy interlayer as a nonmagnetic interlayer -- V, Nb, Ta, Cr, Mo, W, Mn, nickel, Cu, C, Si, and germanium -- each 8at% -- the thin film added and formed, and Pd, Pt, Ag, Au, Ru, Os, Rh and Ir -- each 3at% -- the thin film added and formed was used. Furthermore, with DC magnetron sputtering equipment, the protection lubricating layer 24 and 24' which consist of B by the substrate temperature of 100 degrees C, 1.3Pa of Ar gas pressure, and DC injection power 3 W/cm2 were formed 20nm of thickness, and the magnetic disk was produced.

[The example 1 of a comparison]

Except having used the nonmagnetic interlayer 13 in an example 1, and 13' as Ti single phase film, it is the same configuration as an example 1, and the same spatter conditions, and the magnetic disk was produced.

[The example 2 of a comparison]

Except having used the nonmagnetic interlayer 22 in an example 2, and 22' as Ti single phase film, it is the same configuration as an example 2, and the same spatter conditions, and the magnetic disk was produced.

[The example 3 of a comparison]

Except having removed the nonmagnetic middle class 13 in an example 1, and 13', it is the same configuration as an example 1, and the same spatter conditions, and the magnetic disk was produced.

[The example 4 of a comparison]

Except having removed the nonmagnetic middle class 22 in an example 2, and 22', it is the same configuration as an example 2, and the same spatter conditions, and the magnetic disk was produced.

The measurement result of the c-axis stacking tendency of the Co-Cr magnetic layer in the magnetic disk obtained by the above-mentioned examples 1 and 2 and the examples 1, 2, 3, and 4 of a comparison and magnetic properties is shown in the 1st table. Here, deltatheta50 of a Co-Cr magnetic layer is half a locking curve of 002 reflection of Co-Cr.

	第	1	表	
基板	非磁性中間層	Δθ 50(度)	Hc⊥(0e)	備考
NiP/AI/強化ガラス	Ti-V	5.7/4.8	800/845	実施例1/実施例2
	Ti—Nb	5,3/4,2	720/830	
	Ti —Ta	5.0/4.1	860/910	
	Ti —Cr	5.4/4.4	730/900	
	Ti—Mo	5,8/5,0	650/710	
	Ti—₩	5,7/4.9	670/730	
	Ti — Mn	5.6/4.7	720/740	
	Ti-Ni	6, 1/5, 5	. 530/610	
	Ti —Cu	5,5/4,6	650/730	
	Ti -C	5,9/5,4	580/620	
	Ti —Si	5,7/5,3	630/700	
	Ti —Ge	5.8/5.4	600/670	
	Ti —Pd	7,2/6,7	830/880	-
	Ti —Pt	5,2/4,5	850/890	
	Ti-Ag	5.7/4.8	670/710	
	Ti — Au	5,6/4,6	690/730	
	Ti—Ru	5,5/4.7	700/750	
	Ti−0s	5,5/4,6	680/720	
	Ti —Rh	5.6/4.7	710/760	]
	Ti-lr	5.7/4.9	650/680	
Ni -P/A1	Ti	11.8	703	比較例 1
強化ガラス	Тi	8.2	1140	比較例 2
Ni -P/Ai	なし	8.0	325	比較例3
強化ガラス	なし	9.0	380	比較例 4

=

It is range of prices, and a stacking tendency is so high that deltatheta50 is small. Hc\*\* is the coercive force when impressing a field perpendicularly to a substrate side. Surfacing spacing 0.24micrometer from a magnetic layer front face estimated the record reproducing characteristics of each above-mentioned medium by the Mn-Zn ferrite ring head. Here, D50 is track recording density in case a solitary-wave playback output becomes half. According to this invention, compared with the examples 3 and 4 of a comparison which prepare neither the examples 1 and 2 only of a comparison which prepared Ti single phase film as a nonmagnetic interlayer, nor a nonmagnetic interlayer, the coercive force and C shaft stacking tendency of a Co-Cr magnetic layer are improving sharply in the examples 1 and 2 like [ it is \*\*\*\*\* from these results and ]. Good record reproducing characteristics were acquired corresponding to this. When Nb and Ta are especially added to Ti, the best c-axis stacking tendency is acquired. Moreover, this effectiveness was accepted also when substrates, such as a tempered glass substrate, a ceramic substrate, and a plastics coat aluminum alloy substrate, were used. Compared with the magnetic disk drive using the disk according [ equipment capacity ] to the example of a comparison, equipment capacity was large more than twice, the mean time of the magnetic disk drive using the disk by this invention until equipment will be further downed by sliding etc. was also long single or more figures, and it was especially good. [Example 3]

The nonmagnetic substrate 32 with which 31 changes from the tempered glass which set detailed irregularity to 5nm at the circumferencial direction to a front face in <u>Fig. 3</u>, and 32' the center line average side granularity of owner Perilla frutescens (L.) Britton var. crispa (Thunb.) Decne. High permeability magnetic layers, such as Co-Zr-Mo, Fe-nickel, Mo-Fe-nickel, and a Fe-Co-aluminum-Si alloy, Hexagonal system magnetism alloy layer [ to which nonmagnetic

interlayer / to whom 33 and 33' changes from Ti radical alloy / 34, and 34' changes from Co-Cr etc. J, 35, and 35' is a protection lubricating layer which consists of C, B, B4C, Si-C, etc., and each is formed as shown below. On the tempered glass substrate 31, with DC magnetron sputtering equipment The substrate temperature of 150 degrees C, 0.6Pa of Ar gas pressure. 20at%Fe-nickel or the Co-Zr-Mo quantity permeability magnetic layer 32, and 32' are formed 500nm of thickness on the spatter conditions of DC injection power 6 W/cm2. Ti radical alloy interlayer of the following [ the spatter conditions that it is same on it ] Thickness 2, 5, and 10 and after forming 20nm and considering as the nonmagnetic interlayer 33 and 33', 5at%Ta-16at% Cr-Co or the 5at%Zr-15at%Cr-Co magnetic layer 34, and 34' were formed 200nm of thickness on the same spatter conditions, here -- as a nonmagnetic interlayer -- Ti radical alloy interlayer --V, Nb, Ta, Cr, Mo, W, Mn, nickel, Cu, C, Si, and germanium -- each 11at% -- the thin film added and formed, and Pd, Pt, Ag, Au, Ru, Os, Rh and Ir -- each 6at% -- the thin film added and formed was used. Furthermore, with DC magnetron sputtering equipment, the protection lubricating layer 35 and 35' which consist of B by the substrate temperature of 100 degrees C, 1.3Pa of Ar gas pressure, and DC injection power 5 W/cm2 were formed 20nm of thickness, and the magnetic disk was produced. The about 2-time high output was obtained compared with the record reproducing characteristics of the magnetic disk which produced this magnetic disk by the approach of examples 1 and 2 as a result of evaluating record reproducing characteristics using the vertical-type magnetic head, and good record reproducing characteristics were shown especially. Moreover, any disk showed the good CSS-proof (contact start stop) property beyond 30k times, and was excellent also in sliding-proof nature.

In addition, as said hexagonal system magnetism alloy layer, even if it used hexagonal system magnetism alloy layers, such as Co-V, Co-Mo, Co-W, Co-Re, Co-Ti, Co-Sm, Co-Mn, Co-Pd, Co-Pt, Co-Fe, Co-O, Co-Cr-Rh, Co-Cr-Ru, Co-Cr-Ti, Co-Cr-Hf, Co-Cr-Pt, Co-Cr-Pd, Co-Ti-Ta, Co-Mo-Ta, Co-W, and Ru, in addition to the Co-Cr magnetic layer, there was same effectiveness. Furthermore, in an example 1, an example 2, and an example 3, since sliding-proof nature will improve if 2nm or more 8nm or less of lubricating layers, such as perfluoro polyether system polarity lubricant, is formed on Fig. 1, Fig. 2 and protection lubricating layer [ of a 3rd / \*\* / Fig. R> Fig. ] 15, 15', 24, 24' and 35, and 35', it is still more desirable.

[Effect of the Invention]

In the vertical-magnetic-recording medium which has Ti radical alloy interlayer between a hexagonal system magnetism alloy layer and a nonmagnetic substrate by this invention as explained above By using the thin film which added at least one sort of elements out of the group which consists of V, Nb, Ta, Cr, Mo, W, Mn, nickel, Pd, Pt, Cu, Ag, Au, C, Si, germanium, Ru, Os, Rh, and Ir to said Ti radical alloy interlayer A hexagonal system magnetism alloy interlayer's c-axis stacking tendency and magnetic properties can be improved sharply, and the magnetic disk and equipment which have good record reproducing characteristics and dependability can be offered.

[Translation done.]

### (19)日本国特許庁 (JP)

# (12) 特 許 公 報 (B 2)

(11)特許番号

# 第2669529号

(45)発行日 平成9年(1997)10月29日

(24)登録日 平成9年(1997)7月4日

(51) Int.Cl.<sup>6</sup>

識別記号

庁内整理番号

FΙ

技術表示箇所

G11B 5/66

G11B 5/66

請求項の数11(全 7 頁)

(O1) UISSAN EI	44 ISSETTION 000F0	(70) 4+ 3+4-4-4	00000000
(21)出願番号	特願昭63-89058	(73)特許権者	999999999
			株式会社日立製作所
(22)出願日	昭和63年(1988) 4月13日		東京都千代田区神田駿河台4丁目6番地
		(72)発明者	松田 好文
(65)公開番号	特開平1-263910		東京都国分寺市東恋ケ窪1丁目280番地
(43)公開日	平成1年(1989)10月20日		株式会社日立製作所中央研究所内
		(72)発明者	城石 芳博
		•	東京都国分寺市東恋ケ窪1丁目280番地
			株式会社日立製作所中央研究所内
		(72)発明者	<b>菱山</b> 定夫
			東京都国分寺市東恋ケ窪1丁目280番地
			株式会社日立製作所中央研究所内
	•	(74)代理人	弁理士 小川 勝男 (外1名)
		審査官	蔵野 雅昭
			最終頁に続く

## (54) 【発明の名称】 垂直磁気配録媒体および磁気ディスク装置

#### (57) 【特許請求の範囲】

【請求項1】六方晶系の磁性合金層と非磁性基板との間に少なくともTiの他にV,Nb,Ta,Cr,Mo,W,Mn,Ni,Pd,Pt,Cu,Ag,Au,C,Si,Ge,Ru,Os,Rh及UIrから成る群から選ばれた少なくとも1種の元素を含むTi基合金中間層を有することを特徴とする垂直磁気記録媒体。

【請求項2】上記Tiの他にV.Nb,Ta,Cr,Mo,W,Mn,Ni,Pd,Pt,Cu,Ag,Au,C,Si,Ge,Ru,Os,Rh及びIrから成る群から選ばれた少なくとも1種の元素を含むTi基合金中間層と非磁性基板との間にさらに高透磁率磁性層を設けることを特徴とする特許請求の範囲第1項に記載の垂直磁気記録媒体。

【請求項3】前記Ti基合金中間層に対して、V,Nb,Ta,Cr,Mo,W,Mn,Ni,Cu,C,Si及びGeから成る群から選ばれた少なくとも1種の元素を含みその組成比が総量で1at%以

上25at%以下であることを特徴とする特許請求の範囲第 1項または第2項に記載の垂直磁気記録媒体。

【請求項4】前記Ti基合金中間層に対して、Nb,Taのうち何れか1種の元素の組成比が5at%以上20at%以下であることを特徴とする特許請求の範囲第3項に記載の垂直磁気記録媒体。

【請求項5】前記Ti基合金中間層に対して、Ru,0s,Rh,Ir,Pd,Pt,Ag及びAuから成る群から選ばれた少なくとも1種の元素を含みその組成比が総量で0.1at%以上10at%以下であることを特徴とする特許請求の範囲第1項または第2項に記載の垂直磁気記録媒体。

【請求項6】前記Ti基合金中間層に対して、特にRu,Os,Rh,Ir,Pd,Pt,Ag及びAuから成る群から選ばれた少なくとも1種の元素を含みその組成比が総量で1at%以上7.5at%以下であることを特徴とする特許請求の範囲第5項に

2

3

記載の垂直磁気記録媒体。

【請求項7】前記六方晶系の磁性合金層をCo組成比が50 at%以上のCo基合金薄膜とすることを特徴とする特許請求の範囲第1項ないし第6項に記載の垂直磁気記録媒体。

【請求項8】前記Co基合金薄膜が、Cr.V.Mo.W.Ti.Mn.Re.Sm.Fe及びOから成る群から選ばれた少なくとも1種の元素を含みその組成比が総量で0.1at%以上25at%以下であることを特徴とする特許請求の範囲第7項に記載の垂直磁気記録媒体。

【請求項9】前記Co基合金薄膜が、Zr,Ti,Hf,Ta,Ru,Rh,Pd及びPtから成る群から選ばれた少なくとも1種の元素を含みその組成比が総量で0.1at%以上15at%以下含むことを特徴とする特許請求の範囲第7項または第8項に記載の垂直磁気記録媒体。

【請求項10】前記非磁性基板として、強化ガラス基板、Ni-Pメッキ層付きA1合金基板及びセラミックス基板の何れか1種を用いることを特徴とする特許請求の範囲第1項ないし第9項に記載の垂直磁気記録媒体。

【請求項11】特許請求の範囲第1項ないし第10項に記 20 載の垂直磁気記録媒体を用いた磁気ディスク装置。

#### 【発明の詳細な説明】

#### [産業上の利用分野]

本発明は次世代の超高密度記録が可能な媒体として注目されている垂直磁気記録媒体に係わり、特に高密度記録に好適な磁気ディスク装置用媒体に関する。

#### [従来の技術]

従来、超電密度記録用の垂直磁気記録媒体として、特 公昭57-17282号に記載のように、磁性膜としてCo-Cr 合金薄膜を用いた媒体が提案されている。垂直磁気記録 用媒体の形成法としては蒸着法、スパッタリング法、メ ッキ法などがある。最近、記録密度の向上がますます要 求されてきており、そのため特開昭49-74912号公報、 特開昭58-14318号公報のようにCo-Cr合金薄膜と基板 との間にTi中間層を設け、結晶性を制御して高い c 軸配 向性を得る提案がなされている。また、特開昭58-1336 24号公報には、Co-Cr合金との熱膨張率の差を少なく し、記録時と再生時におけるトラックずれを小さくする ために垂直磁気記録媒体用の基板として、Ti基合金を用 いることや、さらに特開昭62-143227.62-143228.62-143229号公報には下地層としてTi合金中間層を設け、さ らに磁性層の結晶粒径を10~300nmとすることで、真空 蒸着法でも高い保磁力の媒体を得ようとする提案もなさ れている。

#### [発明が解決しようとする課題]

しかし、これらの発明はほとんど蒸着法、イオンプレーティング法などによる、磁気記録用テープやフレキシブルディスクに関するものであり、強化ガラスやNi-PメッキA1合金のように平坦で硬い材料からなる基板を用いるコンピュータ用リジッドディスクにおいてはあまり

検討されていない。そこで本発明者らは、まずNi-Pメ ッキA1合金基板上に直接Co-Cr合金をRFスパッタリング 法で形成し特性を評価してみた。ここでCrの組成は5~ 25at%, 成膜時の基板温度:室温~150℃,Arガス圧3~ 30mTorr {0.4~4Pa} , 投入電力密度 1~10W/cm², 膜厚6 0~250nmとした。いずれのCo-Cr膜も垂直方向の保磁力 は3000e以下と低く、しかもCo-Crの c 軸配向性の分散 Δθ50も10°以上と悪く、良好な垂直磁化膜は得られな かった。強化ガラス基板についても同様であった。これ はNi-P基板や強化ガラス基板は通常の有機系基板に比 べて表面エネルギーが高く六方晶のCo-Crの最密面であ るc面の高配向が阻害されるためである。一般に垂直磁 気記録用の磁性膜において高い記録密度を達成するに は、垂直方向の保磁力を高めると共に垂直磁化膜の結晶 軸(c軸)を膜面に垂直に配向させることが特に重要で あることが知られている。

本発明の目的は、高い密着性が得られるスパッタリング法などにより磁気記録用テープやフレキシブルディスクだけでなく、強化ガラスや金属材料などの硬質基板を用いるリジッドディスクとしても良好な結晶配向性、磁気特性及び密着性を有する垂直磁化膜を形成し、高記録密度が可能で信頼性の高い磁気記録媒体を提供することにある。

#### [課題を解決するための手段]

六方晶系の磁性合金層と強化ガラス基板、Ni-Pメッ キ層付きA1合金基板及びセラミックス基板等の非磁性基 板との間にSi基合金、Ti基合金、Zr基合金等の種々の非 磁性中足層を設けた媒体をスパッタ法などで形成し、そ の媒体の結晶学的特性及び磁気的特性、記録再生特性さ らに密着性、耐食性等の信頼性などを鋭意検討した本発 明者等の研究によれば、上記の目的は前記非磁性中間層 としてTiの他にV,Nb,Ta,Cr,Mo,W,Nm,Ni,Pd,Pt,Cu,Ag,A u,C,Si,Ge,Ru,Os,Rh及びIrから成る群から選ばれた少な くとも1種の元素を含むTi基合金中間層を用いることに より達成される。このTi基合金中間層に対して、V.Nb.T a,Cr,Mo,W,Mn,Ni,Cu,C,Si,Geから成る群から選ばれた少 なくとも1種の元素を含みその組成比は総量でlat%以 上25at%以下が望ましく、Pd.Pt.Ag.Au.Ru.Os.Rh及びIr から成る群から選ばれた少なくとも1種の元素を含みそ の組成比は総量で0.1at%以上10at%以下とすることが 望ましい。さらに、前記Ti基合金中間層に対して、特に Nb, Taのうち何れか 1 種の元素の組成比が5at%以上20at %以下であることがより望ましく、Pd,Pt,Ag,Au,Ru,Os, Rh及びIrから成る群から選ばれた元素を含みその組成比 が総量でlat%以上7.5at%以下とすることがより望まし い。また、前記六方晶系の磁性合金層としてはCoを50at %以上含み、さらにCr,V,Mo,W,Ti,Mn,Re,Sm,Fe及びOか ら成る群Xから選ばれた少なくとも 1 種の元素を総量で 0.1at%以上25at%以下含むCo-X2元合金薄膜を用いる と、磁気特性の上でより効果的である。さらに、前記六

方晶系の磁性合金層として、Zr,Ti,Hf,Ta,Ru,Rh,Pd及びPtから成る群Yから選ばれた少なくとも 1 種の元素を総量で0.1at%以上15at%以下含むCo-Y2元合金薄膜を用いてもよい。前記Co-X2元合金にさらに前記Yから選ばれた少なくとも 1 種の元素を総量で0.1at%以上25at%以下含むCo-X-Y3元合金薄膜を用いると耐食性の上でより効果的である。

### [作用]

上記手段は以下の作用による。これまで、特開昭58-159225,59-22236,59-22225,59-33628号公報などにポ リイミド等の耐熱性基板上にTi薄膜を介してCo-Cr薄膜 を形成することで、Co-Cr磁性膜の結晶配向性を高める ことが知られている。そこでまず、Ni-P基板に対して も同様の効果が得られるか検討した。すなわち、Ni-P をメッキし、その表面を鏡面研磨したA1合金基板上に、 RFスパッタリング法で基板温度:室温~200℃,Arガス圧 3~30mTorr {0.4~4Pa} , 投入電力密度 1~10W/cm²と して膜厚20~600nmのTi中間層を形成し、さらに連続し て膜厚60~250nmのCo-Cr合金磁性層を形成し、特性を 評価した。Cr組成については5~25at%まで変えて検討 した。その結果、Ti中間層を設けることによりCo-Crの 保磁力は3000e以上に高くなり、基板温度が高い程高い 保磁力が得られたが、いずれの膜においてもCo-Crの c 軸配向性の分散を示すΔθ50は10°以上と大きく、良好 な c 軸配向性を示さなかった。蒸着法では15°以上とさ らに低い配向性の膜しか得られず、強化ガラス基板でも Ni-P基板と同様の結果しか得られなかった。一方、ポ リエチレンテレフタレート, ポリイミド, ポイアミド等 の有機系基板に対してはいずれの方法でも  $\Delta$   $\theta$  50 は約5 と高いc軸配向性を有する膜が得られた。これは、Ni 30 - P 基板や強化ガラス基板は有機系基板に比べて表面エ ネルギーが高く、Ti中間層の最密面であるc面の配向も 阻害されてしまうためである。そこで本発明者らは、ま ず中間層の配向性を高めることについて上記成膜条件 で、Tiに4A,5A,6A,7A,8,1B,2B,3B,4B,5B,6B族の元素を 添加した中間層を成膜、特性評価することで鋭意検討す ることにした。その結果、いずれの成膜条件において も、Tiの他に、V,Nb,Ta,Cr,Mo,W,Mn,Ni,Pd,Pt,Cu,Ag,A u,C.Si,Ge,Ru,Os,Rh及びIrから成る群から選ばれた少な くとも 1 種の元素を含むTi基合金中間層は、Ti単相膜に 比べて、非磁性基板上に垂直に c 軸が配向し易いことが RHEED, X線回折法などにより明らかになった。実際、第 4図(a)にはNi-P基板上に形成した、TiにNb,Taに 添加したTi基合金中間層とその上に形成したCo-Cr磁性 合金層の c 軸配向性を示すが、Ti基合金中間層に対して Nb, Taの何れか 1 種の組成比が5at%以上20at%以下のと きに、Ti-Nb,Ti-Ta合金中間層及びCo-Cr磁性合金層 の002反射のΔθ50が小さく、特にCo-Cr磁性合金層の c 軸配向性が高いことが分かる。Nb-Ta合金やV.Cr.Mo. W,Mn,Ni,Cu,C,Si及びGeから成る群から選ばれた元素の

1種をTiに添加した場合やこれらの組み合わせから成る 合金も同様の添加量で同じ効果が認められた。ここでTi にNb,Taのいずれか1種を添加した膜は、V,Cr,Mo,W,Ni, Cu,C,Si及びGeの1種を添加した場合に比べて耐食性が 高く中間層としては特に好ましい。また、Ti基合金中間 層にNb.Taの何れか 1 種を5at%以上20at%以下添加した 場合に最も高い c 軸配向性が得られた。さらに、第4図 (b)には、TiにPt,Pdを添加したTi基合金中間層上に 形成したCo-Cr磁性合金層の c 軸配向性を示すが、Ti基 合金に対してPt,Pdの何れか1種の組成比がlat%以上7. 5at%以下のときに、特にCo-Cr磁性合金層の c 軸配向 性が高いことが分かる。Ru,Os,Rh,Ir,Ag及びAuから成る 群から選ばれた元素の少なくとも1種をTiに添加した場 合やTiにRu,Os,Rh,Ir,Ag,Au,Pt,Pdの合金を添加した場 合にも同様の添加量で同じ効果が認められた。ここで、 TiにV,Nb,Ta,Cr,Mo,W,Mn,Ni,Cu,C,Si,Geを添加したTi基 合金系に比べて、これらの合金系は高価ではあるが耐食 性が高く、信頼性の面ではより好ましい。いずれにせ よ、このように、V.Nb.Ta.Cr.Mo.W.Mn.Ni.Pd.Pt.Cu.Ag. Au,C,Si,Ge,Ru,Os,Rh及びIrから成る群から選ばれた少 なくとも1種の元素をTiに添加することにより、Ti基合 金中間層としての c 軸配向性が向上するのは、Ti基合金 のバルクの状態図においてTiの六方晶相を保持できるV. Nb, Ta, Cr, Mo, W, Mn, Ni, Pd, Pt, Cu, Ag, Au, C, Si, Ge, Ru, Os, R h及びIr等の固溶限が非常に小さく、したがって、V.Nb. Ta,Cr,Mo,W,Mn,Ni,Pd,Pt,Cu,Ag,Au,C,Si,Ge,Ru,Os,Rh及 びIr等の成分はTi基合金中間層の柱状結晶の粒界に偏析 し易く、そのために最密面である c 面が基板面に平行に 成長し易いためである。ただし一般にスパッタリング法 等で成膜した薄膜状態での状態図は組成の絶対値、相状 態等はバルクの状態図とは著しく異なることが通常で、 バルクの状態図だけから逆に本効果を予想することは困 難であり、既に説明したような詳細な実験が必要であ る。さらにCo-Cr,Co-V,Co-Mo,Co-W,Co-Re,Co-Ti. Co-Sm, Co-Mn, Co-Ta, Co-Zr, Co-Hf, Co-Pd, Co-Pt, C o-Fe,Co-O,Co-Cr-Rh,Co-Cr-Ru,Co-Cr-Ta,Co-C r-Zr,Co-Cr-Pt,Co-Cr-Pd,Co-Cr-Ti,Co-Cr-Hf, Co-Ti-Ta等の六方晶系磁性合金は上記Ti基合金と格子 定数が近いため、エピタキシャル的に六方晶系磁性合金 層がTi基合金上に成長し易く、上記六方晶系磁性合金か ら成る、高い c 軸配向性を示す垂直磁化膜が得られるこ とになる。ここでCo基3元合金はCo基2元合金に比べて 耐食性が高いので好ましい。

次に本発明より成る高配向垂直磁化膜の磁気特性,記録再生特性について述べる。TiにそれぞれNb,Ta及びPt,Pdを添加したTi基合金中間層上に形成したCo-Cr磁性合金層の磁気特性を第4図(a),第4図(b)に示した。Co-Cr磁性合金層のc軸配向性が高いTi基合金組成で、垂直磁気異方性が高く角形比が大きい等の優れた垂直磁気特性が得られた。また、V.Cr.Mo,W.Mn,Ni,Cu,Ag,

7

Au, C, Si, Ge, Ru, Os, Rh及びIrから成る群から選ばれた少 なくとも1種の元素をTiに添加した場合も同様に、Co基 磁性合金層のc軸配向性が高いTi基合金組成で、優れた 垂直気性特性が得られた。特に、前記六方晶系の磁性合 金層として、Cr,V,Mo,W,Ti,Mn,Re,Sm,Fe及びOから成る 群 X から選ばれた少なくとも 1 種の添加元素を総量で0. lat%以上25at%以下含むCo基合金薄膜を用いると、よ り良好な記録再生特性を示し、添加元素量を0.1at%未 満にすると面内記録成分が著しく強くなり、25at%より 多くすると再生出力が著しく低下し、何れも記録再生特 10 性は劣化した。さらに、前記六方晶系の磁性合金層とし て、Zr,Ti,Hf,Ta,Ru,Rh,Pd及びPtから成る群Yから選ば れた少なくとも1種の添加元素を総量で0.1at%以上15a t%以下含むCo基合金薄膜を用いても良好な記録再生特 性を示すが、添加元素量を0.1at%未満にしても、15at %より多くしても、前記と同様に記録再生特性は劣化し た。ここで前記のようにCo-X2元合金にY群から選ばれ た少なくとも 1 種の元素を総量で0.1at%以上25at%以 下添加したCo-X-Y3元合金は、再生出力が若干低下す るが優れた耐食性を示すので応用上より好ましい。以上 20 の効果は蒸着法によっても確認されたが、  $\Delta$   $\theta$  50 は 7  $^{\circ}$ 程度と、効果の大きさはスパッタリング法によるものに 比べて大きかった。これは一般に蒸着法で成膜された膜 は、蒸着粒子の運動エネルギーが0.1~1eVと小さいの で、1桁程度高い運動エネルギーを有する、スパッタリ ング法による膜と比べて膜の配向性や密着性に劣るため である。このようにスパッタリング法による膜は密着性 にも優れているため、耐摺動性等の信頼性の面でも好ま しい。以上の効果はスパッタリング中のガス中にH2 .02. № 等の不純物ガスを1vo1%まで添加してもほぼ同様であ 30 った。

以上の効果により、スパッタリング法などにより非磁性基板上に非磁性中間層として、V,Nb,Ta,Cr,Mo,W,Mn,Ni,Pd,Pt,Cu,Ag,Au,C,Si,Ge,Ru,Os,Rh及びIrから成る群から選ばれた少なくとも1種の元素を含むTi基合金中間層を形成することにより、六方晶系磁性合金層は基板面に対して垂直方向にc軸配向性が高く、優れた垂直磁気特性を示すと共に密着性も高いので、本発明より成る六方晶系磁性合金層を用いることにより、特に優れた記録再生特性及び信頼性を有する垂直磁気記録媒体及び装置40を提供することができる。

#### [実施例]

以下、本発明の実施例を説明する。

# [実施例1]

第1図において、11はA1合金等から成る非磁性基板、12.12′はNi-P.Ni-W-Pもしくはこれらを主たる成分とする合金から成る非磁性メッキ層、13.13′はTi基合金から成る非磁性中間層、14.14′はCo-Crから成る六方晶系磁性合金層、15.15′はC.B.B4C.Si-C.Co3O4.SiO2.Si3N4.W-C.Si-C.Zr-C等から成る保護潤滑層で

あり、それぞれは以下に示すように形成される。外径13 OmmΦ、内径40mmΦ、厚さ1.9mmのA1合金基板11の上に、 膜厚20μmの非磁性12wt%P-Niメッキ層12.12'を形 成した後、表面を円周方向に微細な凹凸を有しその中心 線平均面粗さ10nmになるように鏡面研磨して膜厚を15 μ mとした。この基板上にRFマグネトロンスパッタ装置に より基板温度100℃, Arガス圧0.7Pa, RF投入電力5W/cm²の スパッタ条件で以下のTi基合金中間層を膜厚400nm形成 して非磁性中間層13.13′とした後、同一のスパッタ条 件で21at%Cr-Co磁性層14,14'を膜厚250nm形成した。 ここで、非磁性中間層としてTi基合金中間層には、V.N b,Ta,Cr,Mo,W,Mn,Ni,Cu,C,Si及びGeを各々10at%添加し て形成した薄膜と、Pd.Pt.Ag.Au.Ru.Os.Rh及びIrを各々 5at%添加して形成した薄膜を用いたさらにDCマグネト ロンスパッタ装置により、基板温度100℃.Arガス圧1.3P a,DC投入電力3W/cm²のスパッタ条件でCから成る保護潤 滑層15,15'を膜厚30nm形成し、磁気ディスクを作製し た。

#### [実施例2]

第2図において、21は表面に円周方向に微細な凹凸を 有しその中心線平均面粗さを5nmとした強化ガラス等か ら成る非磁性基板22,22' はTi基合金から成る非磁性中 間層23,23'はCo-Cr等から成る六方晶系磁性合金層、2 4,24′ はC,B,B<sub>4</sub>C,S<sub>1</sub>-C等から成る保護潤滑層であり、 それぞれは以下に示すように形成される。強化ガラス基 板21上にDCマグネトロンスパッタ装置により、基板温度 100℃, Arガス圧0.5Pa, DC投入電力7W/cm²のスパッタ条件 で以下のTi基合金中間層を膜厚200nm形成して非磁性中 間層22,22' とした後、同一のスパッタ条件で20at%Cr -Co磁性層23,23'を膜厚200nm形成した。ここで、非磁 性中間層としてのTi基合金中間層には、V,Nb,Ta,Cr,Mo, W.Mn,Ni,Cu,C,Si及びGeを各々8at%添加して形成した薄 膜と、Pd,Pt,Ag,Au,Ru,Os,Rh及びIrを各々3at%添加し て形成した薄膜を用いた。さらに、DCマグネトロンスパ ッタ装置により、基板温度100℃, Arガス圧1.3Pa, DC投入 電力3W/cm<sup>2</sup>でBから成る保護潤滑層24,24′を膜厚20nm 形成し、磁気ディスクを作製した。

#### [比較例1]

実施例1における非磁性中間層13,13′をTi単相膜とした以外は実施例1と同一構成かつ同一スパッタ条件で、磁気ディスクを作製した。

# [比較例2]

実施例2における非磁性中間層22,22'をTi単相膜とした以外は実施例2と同一構成かつ同一スパッタ条件で、磁気ディスクを作製した。

#### [比較例3]

実施例1における非磁性中間層13,13′を除いた以外 は実施例1と同一構成かつ同一スパッタ条件で、磁気ディスクを作製した。

# [比較例4]

気ディスクにおけるCo-Cr磁性層の c 軸配向性及び磁気

実施例2における非磁性中間層22,22′を除いた以外 は実施例2と同一構成かつ同一スパッタ条件で、磁気デ ィスクを作製した。

上記実施例1,2及び比較例1,2,3,4によって得られた磁

特性の測定結果を第1表に示す。ここで、Co-Cr磁性層 の Δ θ 50 はCo-Crの002反射のロッキング曲線の半

	第	1	表	
基板	非磁性中間層	Δθ50(度)	Hc⊥(0e)	備考
Ni -P/AI/強化ガラス	Ti-V	5.7/4.8	800/845	実施例1/実施例2
	Ti—Nb	5.3/4.2	720/830	
	Ti —Ta	5.0/4.1	860/910	
	Ti —Cr	5.4/4.4	730/900	
	Ti—Mo	5.8/5.0	650/710	
	Ti—₩	5.7/4.9	670/730	
	Ti—Mn	5.6/4.7	720/740	
	Ti —Ni	6, 1/5, 5	530/610	
	Ti —Cu	5.5/4.6	650/730	
-	Ti —C	5,9/5,4	580/620	
	Ti —Si	5,7/5,3	630/700	
	Ti —Ge	5.8/5.4	600/670	
	Ti —Pd	7.2/6.7	830/880	
	Ti —Pt	5, 2/4, 5	850/890	
	Ti — Ag	5.7/4.8	670/710	
	Ti —Au	5.6/4.6	690/730	
	Ti —Ru	5.5/4.7	700/750	
	Ti−0s	5,5/4,6	680/720	
	Ti —Rh	5, 6/4, 7	710/760	
	Ti-lr	5.7/4.9	650/680	_ A_ A_
Ni-P/Al	Ti	11.8	703	比較例1
強化ガラス	Ti	8,2	1140	比較例 2
Ni -P/AI	なし	8.0	325	比較例3

9.0

なし

値幅であり、Δθ50が小さい程配向性は高い。Hc 上は基 板面に対し垂直方向に磁界を印加したときの保磁力であ る。上記の各媒体の記録再生特性はMn-Znフェライトリ ングヘッドにより、磁性層表面からの浮上スペーシング 0.24 μ mで評価した。ここで、D50 は孤立波再生出力が 半分になるときの線記録密度である。これらの結果から 明かなように、本発明によれば、非磁性中間層としての Ti単相膜を設けただけの比較例1,2や非磁性中間層を設 けない比較例3.4に比べて、実施例1.2ではCo-Cr磁性層 の保磁力及びC軸配向性が大幅に向上している。これに 対応して良好な記録再生特性が得られた。特に、TiにN b.Taを添加した場合に最も良好な c 軸配向性が得られて いる。また、本効果は強化ガラス基板、セラミックス基 板、プラスチック被膜Al合金基板等の基板を用いた場合 にも認められた。本発明によるディスクを用いた磁気デ ィスク装置は、装置容量が比較例によるディスクを用い た磁気ディスク装置に比べて装置容量が2倍以上大き く、さらに摺動等により装置がダウンしてしまうまでの 50

強化ガラス

平均時間も1桁以上長く、特に良好であった。 [実施例3]

比較例4

380

第3図において、31は表面に円周方向に微細な凹凸を 有しその中心線平均面粗さを5nmとした強化ガラス等か ら成る非磁性基板32,32′はCo-Zr-Mo,Fe-Ni,Mo-Fe -Ni,Fe-Co-Al-Si合金等の高透磁率磁性層、33,33' はTi基合金から成る非磁性中間層、34,34′はCo-Cr等 から成る六方晶系磁性合金層、35,35' はC,B,B4C,Si-C等から成る保護潤滑層であり、それぞれは以下に示す ように形成される。強化ガラス基板31上にDCマグネトロ ンスパッタ装置により、基板温度150℃,Arガス圧0.6P a、DC投入電力6W/cm2のスパッタ条件で20at%Fe-Niも しくはCo-Zr-Mo高透磁率磁性層32,32'を膜厚500nm形 成し、その上に同一のスパッタ条件で以下のTi基合金中 間層を膜厚2.5.10及び20nm形成して非磁性中間層33.3 3'とした後、同一のスパッタ条件で5at%Ta-16at%Cr -Coもしくは5at%Zr-15at%Cr-Co磁性層34,34′を膜 厚200nm形成した。ここで、非磁性中間層としてTi基合

金中間層には、V.Nb.Ta,Cr,Mo,W,Mn,Ni,Cu,C.Si及びGeを各々11at%添加して形成した薄膜と、Pd.Pt,Ag,Au,Ru,Os,Rh及びIrを各々6at%添加して形成した薄膜を用いた。さらに、DCマグネトロンスパッタ装置により、基板温度100℃,Arガス圧1.3Pa,DC投入電力5W/cm²でBから成る保護潤滑層35,35′を膜厚20nm形成し、磁気ディスクを作製した。この磁気ディスクを垂直型磁気ヘッドを用いて記録再生特性を評価した結果、実施例1及び2の方法で作製した磁気ディスクの記録再生特性に比べて2倍程度の高い出力が得られ、特に良好な記録再生特性を示した。また、いずれのディスクも30k回以上の良好な耐CSS(コンタクト・スタート・ストップ)特性を示し、耐摺動性にも優れていた。

なお、前記六方晶系磁性合金層として、Co-Cr磁性層以外にCo-V,Co-Mo,Co-W,Co-Re,Co-Ti,Co-Sm,Co-Mn,Co-Pd,Co-Pt,Co-Fe,Co-0,Co-Cr-Rh,Co-Cr-Ru,Co-Cr-Ti,Co-Cr-Hf,Co-Cr-Pt,Co-Cr-Pd,Co-Ti-Ta,Co-Mo-Ta,Co-W,Ru等の六方晶系磁性合金層を用いても同様の効果があった。また、さらに実施例1,実施例2及び実施例3において、第1図,第2図及び第3図の保護潤滑層15,15′、24,24′及び35,35′の上にパーフルオロポリエーテル系極性潤滑剤などの潤滑層を2nm以上8nm以下形成すると耐摺動性が向上するのでさらに

好ましい。

#### [発明の効果]

以上説明したように本発明により、六方晶系磁性合金層と非磁性基板の間にTi基合金中間層を有する垂直磁気記録媒体において、前記Ti基合金中間層に対して、V,Nb,Ta,Cr,Mo,W,Mn,Ni,Pd,Pt,Cu,Ag,Au,C,Si,Ge,Ru,Os,Rh及びIrから成る群の中から少なくとも1種の元素を添加した薄膜を用いることにより、六方晶系磁性合金中間層の c 軸配向性及び磁気特性を大幅に改善し、良好な記録再生特性及び信頼性を有する磁気ディスク及び装置を提供することができる。

12

#### 【図面の簡単な説明】

第1図,第2図及び第3図は本発明の実施例の磁気ディスクの断面図、第4図(a)および第4図(b)は本発明の磁気ディスク等における種々の添加元素に対するTi基合金中間層の組成とTi基合金中間層及びCo-Cr磁性合金層のc軸配向性と関係を示す図である。

11,21,31……基板、

12.12' ……非磁性メッキ、

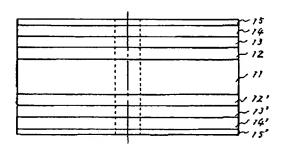
13,13′、22,22′、33,33′……非磁性中間層、

14,14′、23,23′、34,34′……六方晶系磁性合金層、

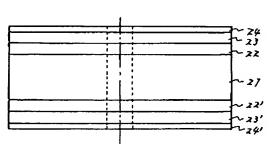
15.15′、24.24′、35.35′……保護潤滑層、

32,32' ……高透磁率磁性層。

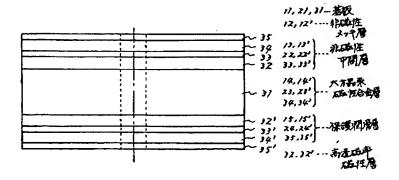
【第1図】



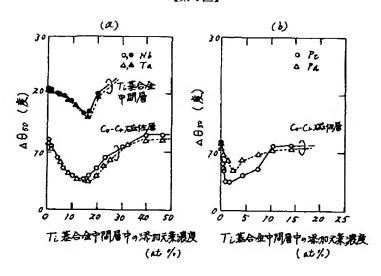
【第2図】



【第3図】



【第4図】



Δθ50···Co-Cr磁性層のC軸配向度

Ho .....保磁力

⊥ ----- 畫直才柯

// ---- 面内方何

# フロントページの続き

(72)発明者 鈴木 博之

東京都国分寺市東恋ケ窪1丁目280番地

株式会社日立製作所中央研究所内

(72)発明者 大野 徒之

東京都国分寺市東恋ケ窪1丁目280番地 株式会社日立製作所中央研究所内 (72)発明者 斎藤 真一郎

東京都国分寺市東恋ケ窪1丁目280番地

株式会社日立製作所中央研究所内

(72)発明者 高木 一正

東京都国分寺市東恋ケ窪1丁目280番地 株式会社日立製作所中央研究所内

(56)参考文献 特開 昭61-113122 (JP, A)

特開 昭63-102024 (JP, A)